

Single- and multi-stage dairy wastewater treatment by vibratory membrane separation processes

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Abstract. Before discharge into sewage or living waters, dairy effluents need to be effectively treated to meet the requirements defined by environmental protection regulations. In addition to the commonly used technologies, membrane separation might offer a novel solution with many remarkable advantages. Although membrane fouling often limits its industrial scale application, module vibration can reduce membrane fouling. In this study, multi-stage membrane separations with ultrafiltration (UF), as pre-filtration, and nanofiltration (NF) were investigated. On the one hand, our aim was to separate the wastewater to reach the cleanest permeate possible, on the other hand to achieve the highest organic content in the smallest volume for further energetic utilization. Firstly, with one-stage separations the effects of Vibratory Shear Enhanced Processing (VSEP) on shear rate, fluxes and rejections were investigated. These tests revealed that vibration has a positive effect on fluxes and rejections. Secondly, two types of multi-stage UF/NF separation experiments were carried out and membrane fluxes, COD rejections and flux decreasing rates were examined. In type 1, permeates of nanofiltered UF permeates achieved the lowest organic load in purified wastewater to meet European environmental threshold limits for living waters. In type 2, concentrates of nanofiltered UF concentrates reached the highest possible volume reduction ratio (VRR) resulting in higher organic content in a smaller volume, which could increase the efficiency of biogas production as an alternative post-treatment for waste management.

Keywords: ultrafiltration; nanofiltration; VSEP; shear rate; multi-stage; dairy wastewater; biogas

1. Introduction

As population growth rate and economic expansion increase, the pollution of our waters is of ever greater concern (Thines *et al.* 2017, Danner *et al.* 2019). Drinking water quality standards are threatened by industrial effluents, such as wastewater produced by the food industry. Production technology of the dairy industry has high water usage throughout the different steps, including the washing of equipment, containers and floor, sanitization, heating and cooling, as well as generating white water, effluents. Dairy effluents usually have high nitrogen, phosphorus, fat, protein and saccharide ratios, containing traces of milk and milk derivatives, and chemicals such as detergents used for cleaning (Karadag *et al.* 2015, Prince *et al.* 2018). Due to this, wastewaters can be characterized by high chemical

oxygen demand (COD) and high biochemical oxygen demand (BOD), which could be converted into several valuable bioproducts, such as biofuels, feed additives, bioplastics or biogas (Chandra *et al.* 2018, Leh-Togi Zobeashia *et al.* 2018). These attributes can lead to serious environmental damage, such as eutrophication, unless proper wastewater treatment is applied before the effluents are discharged into sewage and especially into living waters (Badvipour *et al.* 2016). Therefore, the European Union continuously addresses these environmental issues – mainly by increasing protection regulations, in order to prevent damage to our waters. There are numerous methods available to meet the requirements of these regulations, such as biological or chemical oxidation, trickling filters and anaerobic sludge blanket (UASB) reactors, ion-exchange techniques, anaerobic filters, activated sludge processing or adsorption. Compared to these methods membrane separation is advantageous as it is easily combinable with other technologies and it uses very few chemicals, while it runs on mild operating parameters (Hyun *et al.* 2020, Frappart *et al.* 2008). Ultrafiltration can be a promising method to decrease the organic load of dairy effluents to meet the requirements of discharging into sewers while maintaining high fluxes at a relatively low transmembrane pressure (TMP) (Khosroyar and Arastehnodeh 2018). Nanofiltration (NF) offers higher

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Table 1 Model dairy wastewater characteristics at 50°C

Turbidity	COD	Conductivity	Protein	Viscosity	Density	pH
[NTU]	[mg dm ⁻³]	[mS cm ⁻¹]	[g g ⁻¹]	[m Pas]	[kg m ⁻³]	[-]
221.5	4770 ± 156	0.821 ± 0.03	0.35 ± 0.015	0.37 ± 0.017	983.9 ± 48	7.32 ± 0.34

organic load rejection, but requires higher *TMP* to maintain high fluxes. Unfortunately, membrane fouling still limits the application of membrane processes due to heavy flux decline and decreased membrane lifetime (Wang *et al.* 2020; Bian *et al.* 2000). Earlier results show that the use of vibratory shear enhanced processes (*VSEP*) can efficiently prevent fouling by producing a high shear rate on the surface of the membrane which can alter the cake layer (Akoum *et al.* 2005, 2006; Delaunay *et al.* 2008; Ding and Jaffrin 2014). Multiple researchers have concluded that multi-stage, integrated processes can be very effective for wastewater treatment (Andrade *et al.* 2014; Chen *et al.* 2017; Zhang *et al.* 2014). Luo *et al.* have also measured higher fluxes and experienced less membrane fouling with the multi-stage *UF/NF* process than with the single *NF* method (Luo *et al.* 2011).

In the first part of this study, the feasibility of *VSEP* in dairy wastewater treatment was investigated by processing model dairy effluent with a laboratory mode *VSEP* equipped with different *UF* membranes and an *NF* membrane in order to know the fouling mitigation tendency. The impact of shear rates created by vibration on flux and rejection of *COD*, protein, lactose and salt values were analyzed and compared. Secondly, multi-stage *UF/NF* separation tests were carried out in order to know the possibility to reach the living water discharge thresholds. In this case different vibrational *UF* processes were tested as pre-treatment methods before non-vibrational *NF*. The *UF* permeates were filtered by *NF* as the second step of the treatment. Effects on the permeate flux, flux decreasing rates and rejection values were investigated. To investigate the biogas production tendencies, the *UF* concentrates were also concentrated with *NF*, to concentrate pollutants and organic matter into a smaller volume which is useful for post-treatments.

2. Materials and methods

2.1 Model dairy wastewater

Model dairy wastewater (*ww.*) was prepared from skimmed milk powder (*ww.* concentration of 5 g dm⁻³) (InstantPack, Hungary) and the anionic surfactant cleaning agent Chemipur *CL80* (*ww.* concentration of 0.5 g dm⁻³) (Hungaro Chemicals, Hungary). Characteristics of the *ww.* at 50°C are given in Table 1. These characteristics are similar to real industrial dairy *ww.* (Burak *et al.* 2005).

2.2 Analytical methods

The turbidity of the samples was determined with a Hach2100AN turbidimeter (Hach, Germany). The electric conductivity and pH were measured with a *BVBA* C5010

type multimeter (Consort, Belgium). The samples were tested using closed reflux method for *COD* analysis with an *ET* 108 digester and a *PC* CheckIt photometer (Lovibond, Germany). The lactose and dry content of the samples was measured by a Bentley 150 Infrared milk analyser (Bentley Instruments, USA). The protein and nitrogen contents of the samples were determined by the Kjeldahl method (Foss, Denmark). The viscosity and density of the samples were measured using a vibration viscometer (AND SV-10, Japan) and a portable density meter (Mettler-Toledo Densito 30PX, Switzerland). All of the analytical measurements were repeated three times to calculate an accurate average.

2.2.1 Membrane filtration

Single-step filtrations, the *UF* and *NF* experiments, were carried out using a *VSEP L/P* Series membrane device equipped with a single circular membrane of 0.0503 m² (New Logic Research Inc., USA). Supporting the membrane housing there is a vertical shaft, which acts as a torsion spring and transmits the oscillations of a lower plate, the base which is vibrated by an eccentric drive motor. As a result, the housing containing the membrane oscillates azimuthally with a displacement amplitude adjusted to 2.54 cm on the outer rim at the resonant frequency of 54.1 Hz. The detailed schematic diagram of the *VSEP* system with the calculated shear rates and transmembrane pressure and temperature stepping experiments can be found in our earlier paper (Kertész *et al.* 2017). Separation tests with *VSEP* were carried out at 50±1°C, *TMP* was set to 0.8 MPa for *UF*, and 3 MPa for *NF*. 10 L of feed model wastewater was ultra- or nanofiltered to a retentate volume of 2 L (to volume reduction ratio, *VRR* = 5). Recirculation flow rate was set to 4 *GPM* in every case.

In the multi-stage filtrations, due to the limited recirculation volume of the *VSEP* apparatus, only the *UF* pre-filtration could have been carried out, but for the second-stage another *NF* module had to be used. *NF* was done with a non-vibrational, Uwatech *3DTA* laboratory cross-flow membrane module (Uwatech GmbH., Germany), with the use of flat-sheet 200 Da membranes (in Table 2) with a filtering surface area of 0.0156 m² and 2 MPa *TMP*. In this part, our aim was to reach the highest possible *VRR*, and a smaller volume is more efficient for this purpose. Compared to the 2 L dead volume of *VSEP*, the Uwatech *3DTA* has a significantly smaller, 0.2 L dead volume. 1.6 L of concentrate from the *UF* was used for concentration and processed to a retentate volume of 0.2 L (*VRR*=8). The *VRR* of *UF* was 5 and of *NF* was 8, so the two-stage concentrations resulted in a total *VRR* of 40.

2.2.2 Membranes

The *UF* and *NF* membranes were ordered from *VSEP* Company, but the producers were different (New Logic Research Inc., USA).

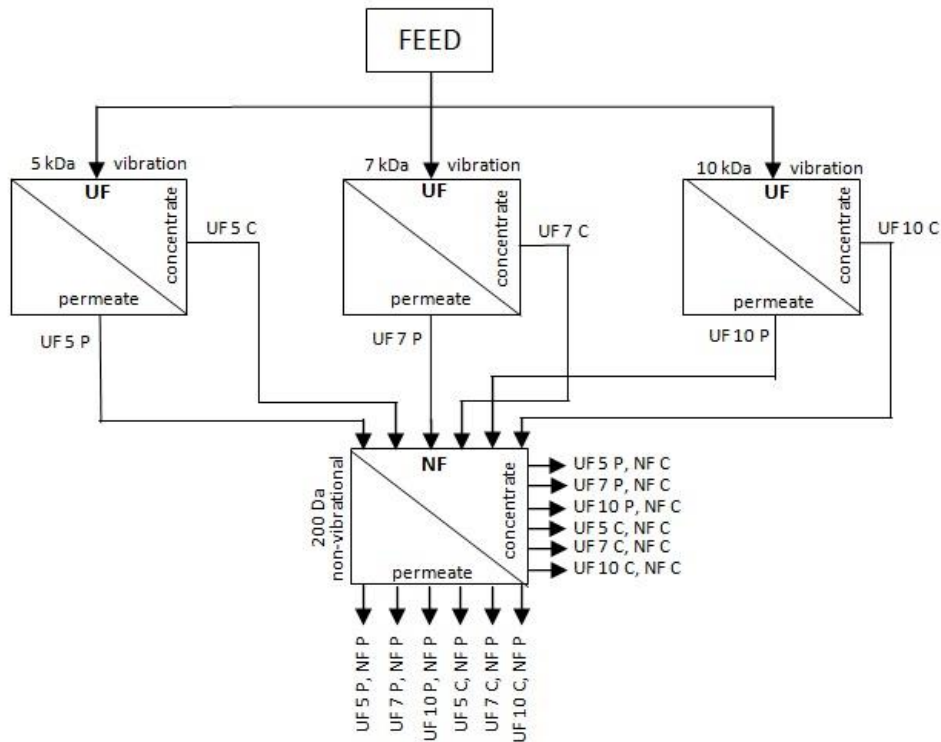


Fig. 1 Multi-stage experiment configuration

Table 2 Characteristics of the membranes used for our experiments

Name	<i>PES-10</i> <i>SYN</i>	<i>PES-5</i> <i>TYVEK</i>	<i>PES-5</i> <i>SYN</i>	<i>NF-3</i>	<i>SR3</i>
Filtration	<i>UF</i>	<i>UF</i>	<i>UF</i>	<i>NF</i>	<i>NF</i>
Material	Polyethersulfone	Polyethersulfone	Polyethersulfone	Thin film composite	Thin film composite
<i>MWCO</i>	10000 Da	7000 Da	5000 Da	240 Da	200 Da
Vendor	Synder	Ultura	Synder	Sepro	Sepro

2.2.3 Anaerobic biogas production tests

Batch mesophilic anaerobic digestion tests were carried out at 37°C for 30 days to determine the biogas yield from the concentrates. Biogas production was detected by the pressure increase method in continuously stirred reactors with volumes of 250 mL equipped by OxiTop-C® measuring heads (WTW, Germany). The temperature was continuously controlled. The pH of the sample was adjusted to 7.2 in the beginning of the experiments. Sludge at 10 g l⁻¹ concentration was used as adaptation inoculum from the local mesophilic biogas digester communal wastewater treatment plant.

2.2.4 Calculated parameters

The selectivity of the membrane, R [%], for a given solute was expressed by the average retention (Eq. (1)):

$$R = \left(1 - \frac{c}{c_0}\right) 100 \quad (1)$$

where c is the average concentration of the solute in the permeate phase, and c_0 is the concentration of the solute in the feed w_w . The volume reduction ratio, VRR [-], was

defined as

$$VRR = \frac{V_F}{V_F - V_P} \quad (2)$$

where V_F is the volume of the feed [m³] and V_P is the volume of the permeate [m³] at any time. The flux decreasing rate (FDR) [%] was expressed by the following eq.:

$$FDR = \left(1 - \frac{J_{WA}}{J_{WB}}\right) 100 \quad (3)$$

where J_{WA} is the water flux of the fouled membrane after the experiment, and J_{WB} is the water flux of the clean membrane before the experiment [m³ m⁻² s⁻¹].

3. Results and discussion

3.1 Single-step experiments

Single-step separation tests using *VSEP* were carried out to investigate the effects of vibration on fluxes and

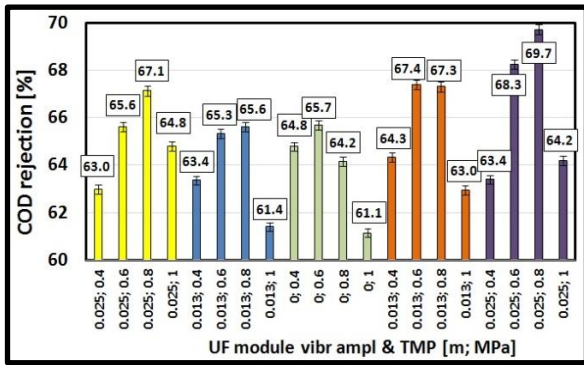


Fig. 2 The effect of vibration on COD rejections (*VSEP UF*: PES 10 kDa; $q_{vr} = 4$ GPM)

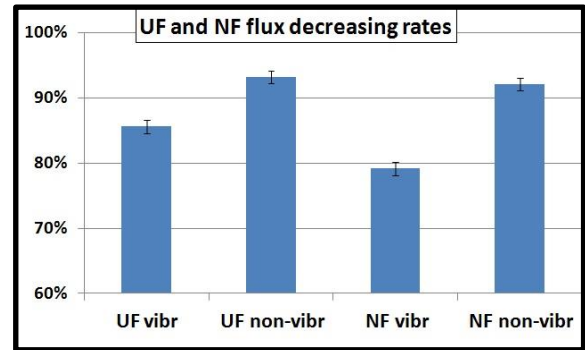


Fig. 3 The effect of vibration on flux decreasing rates (*VSEP UF*: PES 10 kDa; *VSEP NF*: TFC 240 Da, $q_{vr} = 4$ GPM; VRR=5 for UF and 8 for NF)

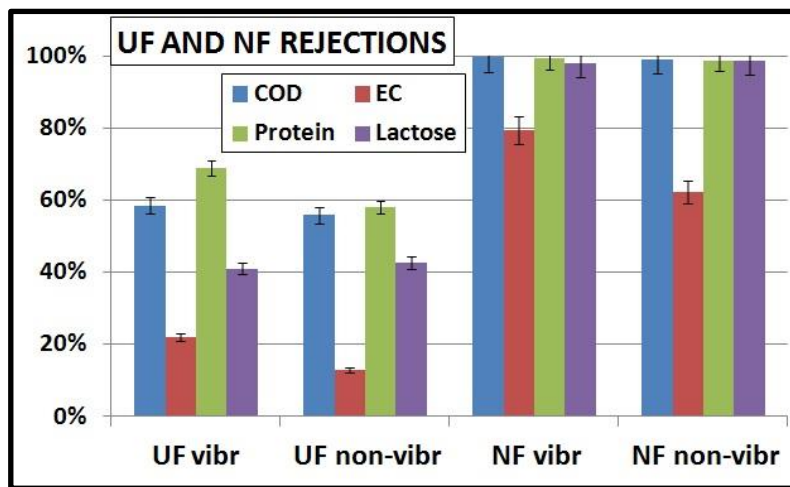


Fig. 4 The effect of vibration on COD rejections (*VSEP UF*: PES 10 kDa; *VSEP NF*: TFC 240 Da, $q_{vr} = 4$ GPM)

rejections. The flux increasing effect of vibration in both *UF* and *NF* was remarkable and less time was needed to achieve the same 5 *VRR* by the end of each process.

The effects of different *TMP* (0.4; 0.6; 0.8 and 1 MPa) at various module vibration amplitudes (0; 0.013 and 0.025 m) on membrane *COD* rejection were studied during ultrafiltration and shown in Fig. 2. It was observed that in all cases *UF* membrane *COD* rejection increased from 0.4 to 0.8 MPa and then decreased suddenly. This was the main reason to use 0.8 MPa *TMP* for further multi-stage experiments in *UF* pre-filtration for the most efficient selectivity.

Flux decline is caused mainly by membrane fouling that can be inhibited by vibration, so calculating flux decreasing rates can be correlated to the effect of module vibration on membrane fouling. Fig. 3 shows that by vibration lessens the extent of membrane fouling in both *UF* and *NF*, which corresponds to the previously discussed effect of vibration increasing flux. In Fig. 4 it is visible that in both *UF* and *NF* chemical oxygen demand (*COD*), electric conductivity (*EC*), protein and lactose rejections could be increased by using vibration. *COD* rejection represents the total organic load rejection, and *EC* rejection shows the salts rejection. Because of its beneficial attributes in further multi-stage experiments vibrational *UF* is used (due to the limited recirculation volume of the *VSEP* apparatus, for the second-stage another *NF* module had to be used).

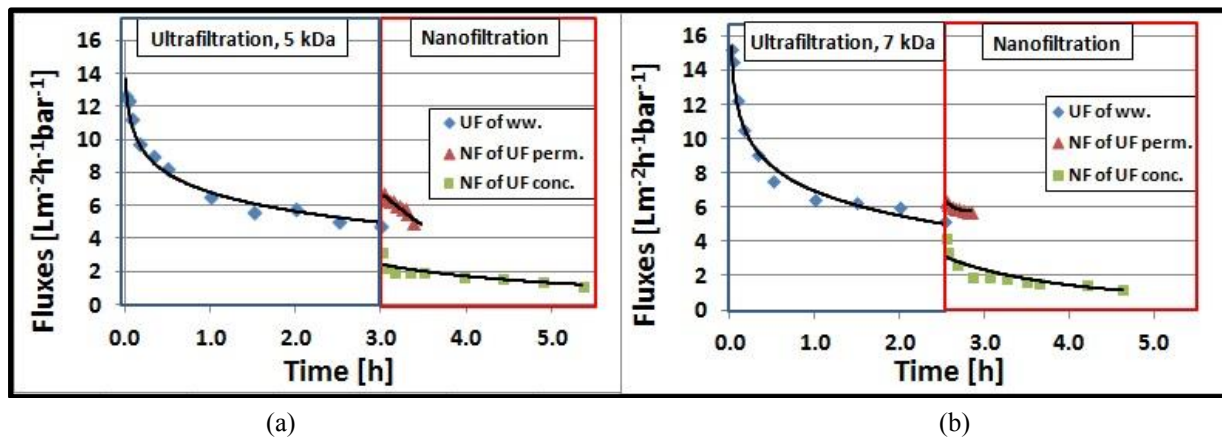
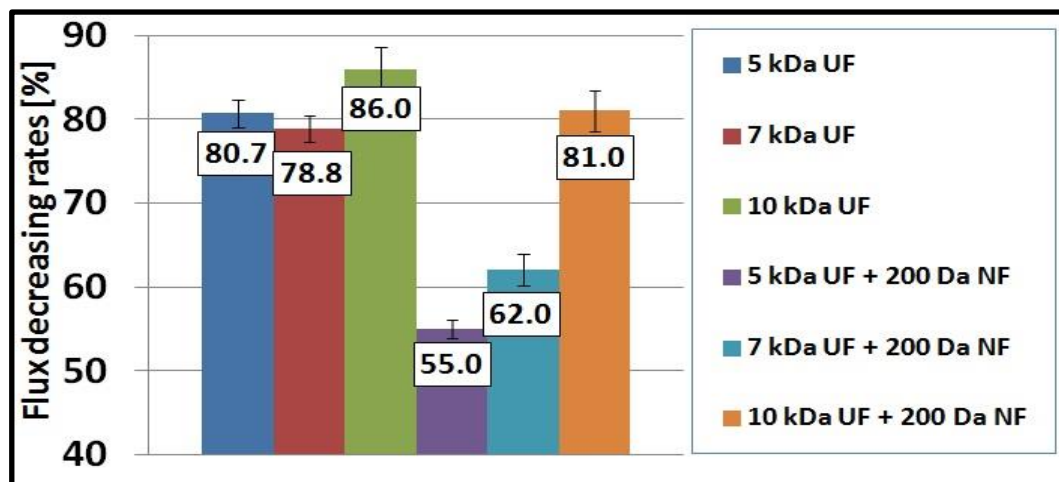
3.2 Multi-stage experiments

To understand the effect of pre-filtration on the *NF* permeates and concentrates, shown in Fig. 5, multi-stage separations were tested with different *UF* pre-filtrations and with the same nanofiltration. Comparing the two different pre-filtrations, 7 kDa *UF* membrane had slightly higher initial fluxes, resulting in shorter filtration time than the 5 kDa *UF* for the same 5 *VRR*. It is noteworthy that the nanofiltrations of the different *UF* permeates practically took the same amount of time to reach the same 8 *VRR*. Regarding the nanofiltration of *UF* concentrates, the one with 7 kDa *UF* pre-filtration was slightly faster than the 5 kDa *UF* concentrate. We assume that the 7 kDa *UF* concentrate was more diluted compared to 5 kDa *UF*, because the higher *MWCO* results in more particles in the permeate, so the refiltration of the concentrate could not foul the membrane as much.

In Table 3, *COD* values of the concentrates and permeates from the multi-stage processes with the smallest *MWCO* membranes (5 and 7 kDa) are given, because these had the highest rejection percentages of the tested membranes. Comparing ultrafiltrations revealed that the 5 kDa *UF* pre-filtration yielded a permeate (*UF 5 P*: based on Fig. 1) with slightly lower *COD* and a concentrate (*UF 5 C*) with higher *COD*, but the difference is minor. After *NF*,

Table 3 Chemical oxygen demand (COD) and membrane rejection values in different process units

5 kDa Ultrafiltration (+ Nanofiltration)			7 kDa Ultrafiltration (+ Nanofiltration)		
Process Unit	COD [mg/L]	Rejection [%]	Process Unit	COD [mg/L]	Rejection [%]
Feed	4770 ± 48	-	Feed	4770 ± 50	-
UF 5 P	1079 ± 11	77.38 ± 0.231	UF 7 P	1103 ± 12	76.88 ± 0.252
UF 5 P, NF P	55 ± 2	98.85 ± 0.042	UF 7 P, NF P	103 ± 4	97.84 ± 0.084
UF 5 P, NF C	4800 ± 46	-	UF 7 P, NF C	5180 ± 52	-
UF 5 C	10070 ± 101	-	UF 7 C	9657 ± 97	-
UF 5 C, NF P	342 ± 4	92.83 ± 0.084	UF 7 C, NF P	156 ± 3	96.73 ± 0.042
UF 5 C, NF C	25006 ± 250	-	UF 7 C, NF C	23640 ± 236	-

Fig. 5 Normalized fluxes of multi-stage filtration test done with different pre-filtration membranes ($VSEP UF: A_{vibr} = 2.54$ cm, 5 kDa/7 kDa PES membrane; Uwatech NF, 200 Da TFC membrane)Fig. 6 Flux decreasing rates in UF and pre-filtered NF experiments ($VSEP UF: A_{vibr} = 2.54$ cm, 5 kDa/7 kDa PES membrane; Uwatech NF, 200 Da TFC membrane)

permeate 'UF 5 P, NF P' had a lower COD compared to the permeate 'UF 7 P, NF P'. Although these values may seem quite small, the 103 mg dm⁻¹ value also meets the European living water discharge thresholds. It was also observed that, different concentration polarization structure and thickness were formed during the experiments in each case. Also, the NF of UF concentrates showed that the concentrate 'UF 5 C, NF C' had higher COD, than concentrate 'UF 7 C, NF C', which indicates that the 5 kDa UF pre-filtration resulted

in a more concentrated retentate, which can lead to more efficient post-treatments, such as biogas production, which will be discussed later in Fig. 7. Therefore we can conclude that whether our goal with the multi-stage process is to have a permeate with the lowest possible COD or to produce a concentrate with the highest possible COD, the 5 kDa UF filtration followed by NF is more efficient.

However, the whole multi-stage process (UF+NF) takes more time when pre-filtration is done with the 5 kDa membrane than with the 7 kDa membrane.

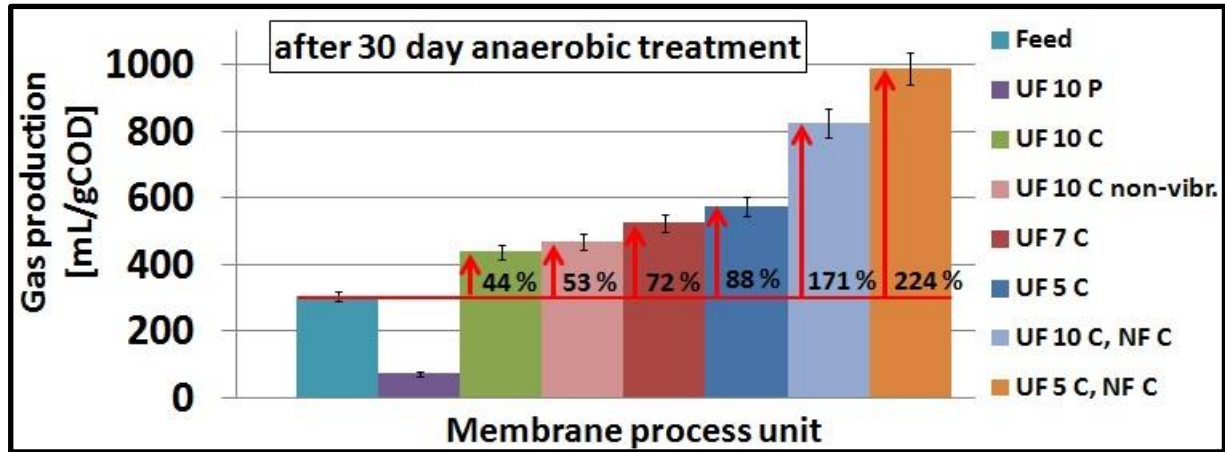


Fig. 7 Biogas production changes from UF and pre-filtered NF concentrates compared to original feed dairy wastewater (process units' names based on Fig. 1)

In Fig. 6, flux decreasing rates in the 5, 7 and 10 kDa *UF* and *UF* pre-filtrations followed by *NF* are compared. In all cases the 10 kDa *UF* has higher *FDR* than the 5 and 7 kDa *UF*, mainly because of its bigger pore size which results in more remarkable fouling. This Fig. also reveals that the *UF* process units alone have much higher *FDR* values than the combined *UF* + *NF* ones. Furthermore, the smaller *MWCO* *UF* membranes decreased the *FDR* of *NF*. Therefore we can conclude that *UF* pre-filtration, with a lower *MWCO* membrane, has a more beneficial effect on nanofiltration *FDR* values.

Biological pre-experiments were done in order to assess the amount of time needed to reach maximum biogas production for the tested feed dairy wastewater and it was observed that 30 days of anaerobic treatment is the optimal time. Therefore, Fig. 7 compares biogas production of the original dairy wastewater and samples from different process units after 30 days. The gas production changed depending on the treatment methods, since the membrane filtered wastewater composition could alter significantly. All of the *UF* concentrates had higher biogas production than the original feed, but the permeate of *UF* exhibited lower production. The volumetric biogas production (ml of produced biogas per organic content of the fermentation broth) changed according to the organic matter content of the concentrate. Fig. 7 shows that the concentrates of smaller *MWCO* membranes had higher biogas production. In terms of the effect of vibration, comparing 'UF 10 C' to 'UF 10 C non-vibr.', a slightly higher biogas production was observed in case of non-vibration. The nanofiltration process after *UF* increased biogas production, compared to original feed ww. by 171 and 224 % in the cases of 'UF 10 C, NF C' and 'UF 10 C, NF C', respectively.

3. Conclusions

In our study dairy wastewater purification was tested by both single- and multi-stage membrane separations. In single-stage experiments by applying vibration, membrane fouling can be reduced, thus, higher fluxes, less flux decline

and slightly higher membrane rejections can be achieved in both ultrafiltration and nanofiltration.

In multi-stage type 1 experiments, the *UF* permeates were nanofiltered, as the *UF* was a pre-filtration process. The aim to meet European environmental *COD* threshold limits for living waters was successfully achieved. In multi-stage type 2 experiments, the *UF* concentrates were nanofiltered, the goal was to achieve the highest possible volume reduction ratio (*VRR*), with high organic content in a smaller volume, which could increase the efficiency of biogas production considerably. Nonetheless, a *VRR* of 40 was reached with certain concentrates, with the highest *COD* measured where the post-treatments are beneficial for biogas production. From biogas production experiments it can be concluded that all of the *UF* concentrates had higher biogas production than the original feed, but permeate of the *UF* produced less. The concentrates of smaller *MWCO* membranes had higher biogas production and nanofiltration after *UF* increased biogas production compared to the original feed ww. almost in average two times.

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